

RAYTHEON

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# Technical Report

## THERMIONIC CATHODE EVALUATION STUDY INTERIM REPORT NO. 1

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RAYTHEON COMPANY  
Microwave and Power Tube Division  
Waltham, Massachusetts

INTERIM REPORT NO. 1  
THERMIONIC CATHODE EVALUATION STUDY

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This report was prepared by F. T. Hill.

This report has been approved by:

G. Freedman  
G. Freedman, Manager  
Materials and Techniques Group

L. L. Clampitt  
L. L. Clampitt, Manager of Engineering,  
Microwave Tube Operation

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## ABSTRACT

Raytheon's Materials and Techniques group is carrying out a study of the life capabilities of three different thermionic cathode types for the Jet Propulsion Laboratory.

This report presents the complete specifications of the cathodes, diode structure, cleaning and exhaust procedures, and preliminary electrical and life testing that was completed in the first interim of this evaluation of thermionic cathodes.

A test diode has been designed and developed for testing electrical characteristics and life expectancy of the three cathode types.

The pore-type dispenser cathode shows that it has the current loading capability of  $0.2 - 1.6 \text{ amperes/cm}^2$  in the temperature ranges of  $950 - 1100^\circ\text{C}$ .

The barium strontium oxide cathode has the current loading capability of  $0.075 - 0.60 \text{ amperes/cm}^2$  in the temperature range of  $750 - 850^\circ\text{C}$ .

The coated powder cathode can be loaded to  $0.138 - 1.10 \text{ amperes/cm}^2$  in the temperature range of  $800 - 900^\circ\text{C}$ .

A life test station has been designed and constructed to operate 48 test diodes with regulated cathode temperatures ranging from  $750 - 1100^\circ\text{C}$  and with constant plate voltage on each socket ranging from  $25 - 200 \text{ volts dc}$ .

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## 1.0 INTRODUCTION

The Materials and Techniques group of Raytheon Microwave Tube Operation is performing a study of the life capabilities of three different types of thermionic emitters for the Jet Propulsion Laboratory, California Institute of Technology.

The life capabilities of the following electron tube cathode types are to be evaluated for a period of 2 years of life testing.

- a. Pore-Type Dispenser Cathode
- b. Standard Barium Strontium Oxide Cathode
- c. Coated Particle Cathode

A life test vehicle has been designed, fabricated, exhausted and tested electrically for conformance with the required cathode current densities.

The diode test vehicle is a glass test structure of the plane parallel type which has a viewing port in the anode for determination of cathode temperatures.

The three cathode types have been designed and manufactured by personnel of the Materials and Techniques group.

A 48-position life test station has been designed, constructed and delivered to the Raytheon Company for use in this study.

The life test station is undergoing stability tests at present. It appears to be very satisfactory for performance of the electrical parameters required for the evaluation of the three types of cathodes.

This report presents the complete specifications of the cathodes, diode structure, cleaning and exhaust procedures, and preliminary electrical and life testing that was completed in the first interim of this evaluation of thermionic cathodes.



## 2.0 THERMIONIC CATHODE DESIGN

The three different thermionic cathodes to be used in this study for evaluation of life capabilities under varying current loading conditions have been designed and have been pretested for conformance with electrical test specifications.

### 2.1 Pore-Type Dispenser Cathode

The pore-type dispenser cathode is a porous tungsten structure impregnated with a molten mixture of barium oxide, calcium oxide and aluminum oxide.

Its thermionic emission mechanism consists of a monolayer of barium metal on oxygen on the tungsten face of the cathode.

Barium metal is generated by chemical reaction of the mixture of barium, calcium, and aluminum oxides with tungsten metal in the pores of the cathode. Barium is continually being generated and diffuses through the pores of the tungsten body to the surface of the cathode, replenishing the evaporating barium on the surface.

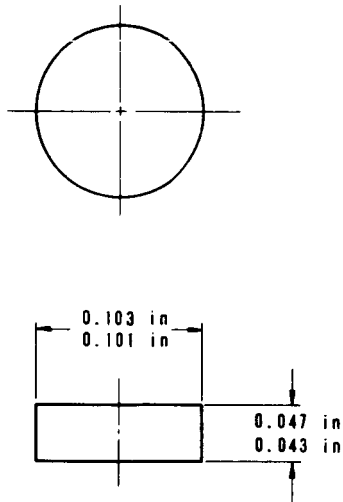
This pore-type dispenser cathode generally operates at 1100°C and has a work function of 1.9 - 2.1 electron volts.

The cathode has capabilities of operating up to 50 amperes/cm<sup>2</sup> under varying conditions of operation, i.e., pulse or dc operation. Emission failure of the cathode occurs when the tungsten emitting face is voided of barium atoms. The emission failure is generally sudden and catastrophic.

Physical dimensions of the cathode to be used in this study are shown in Figures 1 and 2.

The emission pellet is  $\approx$  0.100 in. in diameter and 0.045 in. thick, and has a controlled porosity of 20 - 22%.

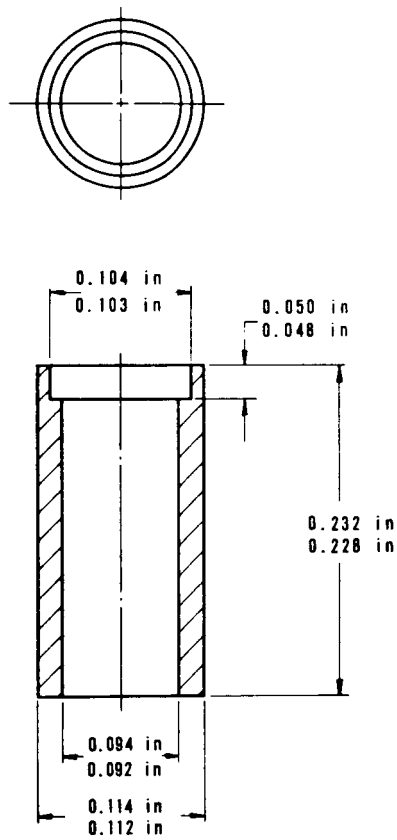
Cu - IMPREGNATED TUNGSTEN  
20 - 22% POROSITY



654810

Figure 1. Emission Pellet

AVC MOLYBDENUM



654811

Figure 2. Sleeve for Emission Pellet

This pellet is prepared by carefully controlling the hydraulic pressing and high temperature sintering of pure tungsten powder. The tungsten powder is capable of passing through a 325-mesh sieve. Chemical analysis of the tungsten powder for residual impurities is as shown in Table I.

TABLE I  
Impurity Analysis of Tungsten Powder

w	-	major constituent
Si	-	0.001 - 0.01%
Fe	-	0.0001 - 0.001%
Cu, B, Ni	-	0.00003 - 0.0003%
Ag, Ca, Mg	-	0.00001 - 0.0001%

After the sintering operation the tungsten slug is impregnated with OFHC copper at high temperature in a hydrogen atmosphere.

The copper-impregnated slug of tungsten metal is then machined to the dimensions shown in Figure 1.

Copper from the machined pellets is removed by vacuum distillation and the porosity of the porous body is determined from the loss of weight of copper.

For this study, 50 pellets of porous tungsten (20 - 22% porosity) will be manufactured from the same lot of tungsten powder, whose impurity analysis is shown in Table I.

The sleeve holder for the emission pellet is machined from a molybdenum rod to the dimensions shown in Figure 2. Sleeves are then cleaned according to the schedule shown in Table II.

TABLE II

## Cleaning of Molybdenum Parts

- a. Degrease in hot tri-chlorethylene
- b. Dip in acid solution (40% nitric acid, 30% phosphoric acid, 30% acetic acid): Parts should be dry before immersing in the acid solution.
- c. Rinse in hot running water twice at 1 minute per rinse
- d. Rinse in acetone
- e. Dry in oven at 110°C
- f. Vacuum fire at 1600°C for 20 minutes

The porous tungsten pellet is polished using fine emery paper to a diameter of 0.100 in. Then it is inserted in the molybdenum sleeve and is peened into the shoulder of the sleeve. A flat surface is achieved by polishing away the rough edges of molybdenum metal.

The mix of barium, calcium, and aluminum oxides is now loaded on the emitting face of the cathode and is melted and absorbed into the pores of the porous tungsten body by high temperature heating in dry hydrogen.

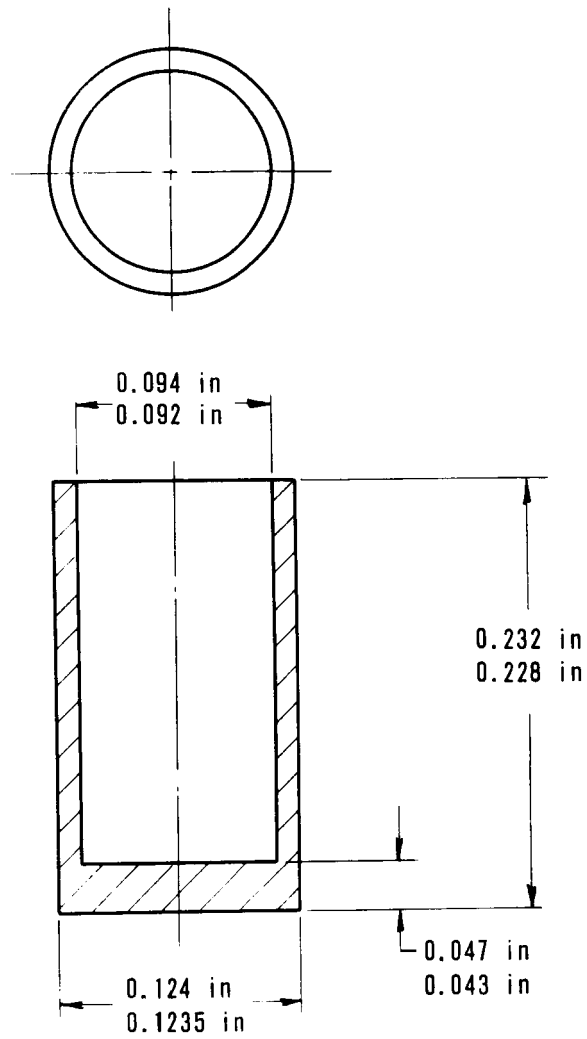
After impregnation of the cathode, it is cleaned by polishing off the excess mixture.

These cathodes are stored in a vacuum desiccator until used.

## 2.2 Standard Barium Strontium Oxide Cathode

The standard barium strontium oxide cathode (better known as the oxide cathode) consists of a metallic body as shown in Figure 3 upon whose emitting face is sprayed a layer of barium and strontium carbonates. Chemical analysis of the metallic body is as shown in Table III.

0.1% ZIRCONIUM - NICKEL



654812

NOTE: USE ONLY NUJOL AS LUBRICANT FOR MACHINING

FIGURE 3. Emission Cathode

TABLE III

Impurity Analysis of 0.1% Zr - Ni

Zr	-	0.092%
Fe	-	0.003%
Mg	-	0.001%
Cu, Si, Mn	-	0.0003%
Cr, Ae	-	0.0001%
Ca	-	0.00001%

The nickel body of the cathode is machined to the appropriate dimensions, using Nujol as a lubricant.

After machining, the cathode is cleaned and preprocessed according to the specifications in Table IV.

The oxide coating used is a standard Raytheon mixture known as C51-3, which consists of a mixture of Baker's RM#3, nitrocellulose, butyl acetate and butyl alcohol. (Baker's RM#3 is a standard barium, strontium carbonate (50-50 molar ratio) used by the vacuum tube industry for over 25 years.) An appropriate weight of Baker's RM#3 is mixed with nitrocellulose as a binder and with a 50-50 mixture of butyl acetate and alcohol as a suspending vehicle.

This mixture is sprayed upon the face of the cathode to give a thickness of 0.002 in. to 0.0025 in. and a coating density of one gram/cm<sup>3</sup>.

These cathodes are stored in a vacuum desiccator until used.

TABLE IV

## Cleaning of 0.1% Zr - Ni Cathodes

- a. Degrease cathodes ultrasonically in a pyrex beaker of fresh (new volume) acetone for 5-6 minutes.
- b. Repeat step (a) for a total of four times using fresh (new volume) acetone each time.
- c. Blow cathodes dry with a stream of filtered nitrogen, pre-purified.
- d. Liquid-hone surface of cathode button for 1-2 minutes to achieve a uniform matte finish using 320 mesh alumina.
- e. Rinse under warm (45-55°C) running tap water for 25-35 seconds.
- f. Wash ultrasonically in a tank of 0.03% Igepal solution for 3-5 minutes.
- g. Rinse under warm (50-59°C) running tap water.
- h. Rinse in deionized water tanks (3 required) for 5-6 minutes in each tank, using next cleanest tank each time.  
Blow parts dry in a stream of filtered nitrogen.
- i. Dry cathodes at 105-115°C for 14-16 minutes in Blue M or Precision Electric Oven using clean dry air or nitrogen atmosphere. Oven must be atomizer clean and produce parts that will pass Atomizer Spray Test.
- j. Fire cathodes in Serpentine or Lindberg /Heviduty Muffle Furnace at 1040-1060°C for 30-31 minutes in wet hydrogen (oxygen-free) atmosphere. After cathodes are cool, store immediately in wide-mouth glass jars with Teflon lined bakelite screw caps.

### 2.3 Coated Particle Cathode

The third cathode involved in this study is the coated particle cathode as described in the publication "Bell Laboratories Record" pp 460 - 461 dated December 1965 and as improved and revised by the Raytheon Company.

This cathode at present is being manufactured and is being tested and used in several vacuum tube devices by the Raytheon Company.

Dimension of the nickel body of the cathode are identical to those shown in Figure 3.

The cathode coating differs from the conventional barium, strontium carbonate coating in that each particle of coating is covered with a thin layer of nickel; Total weight of the nickel amounts to  $1\% \pm 0.3\%$  of total weight of the coating.

A fluid-bed process is used to coat the alkaline earth carbonate particles. A mixture of nickel carbonyl and hydrogen is bubbled through a suspension of the particles in amyl acetate at a temperature of  $120^{\circ}\text{C}$ . Nickel carbonyl decomposes into nickel and carbon monoxide with the nickel depositing on the individual particles of the coating mixture.

When the process has been completed, nitrocellulose is added to the admixture of coated particles and amyl acetate to make a spray mixture.

The 0.1% Zr - Ni cathodes are sprayed to the same specifications as mentioned under Section 2.2. The oxide cathode ( $T = 0.002 \text{ in.} - 0.0025 \text{ in.}$ ,  $D = 1 \text{ gm/cm}^3$ ).

Theoretically, this system should withstand higher current density loading due to the continuous nickel path thru the body of the coating which provides higher electrical conductivity.



Both the oxide and the coated particle cathodes will operate in the temperature range of 750°C - 900°C. The oxide cathode has a work function of 1.0 - 1.2 electron volts and the coated particle cathode has a work function of  $\approx$  1.2 - 1.4 electron volts.

End of life for these cathodes is not catastrophic but gradual under operating conditions.

Fifty 0.1% Zr - Ni cathodes will be made for use with this study.

#### 2.4 Cathode Heater

The heater for the three cathode types is shown in Figure 4. In all cases the same heater will be used.

The pore-type dispenser cathodes will operate at 8.0 - 12.0 Vac and the cathode temperature will range from 900 - 1100°C.

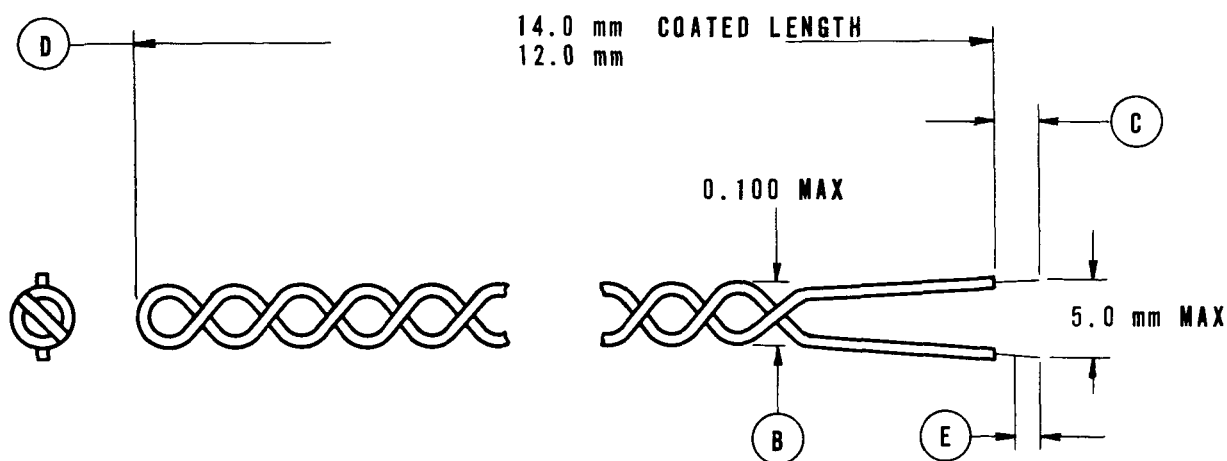
The oxide and the coated particle cathodes will operate at 6.0 - 8.0 Vac and cathode temperatures will range from 750 - 900°C.

It should be noted that the pore-type cathode is smaller in diameter. The diameter is 0.100 in. and the emitting area is 0.05 cm<sup>2</sup>.

The other two cathodes (which have the same nickel sleeve) were made in one piece. Their diameter is 0.125 in. and each has an emitting area of 0.0794 cm<sup>2</sup>.

In all cases, the hole for insertion of the heater is the same (ID = 0.093 in, L = 0.0195 in.).

- A - COATED WIRE O D 0.007 - 0.009 in.
- B - COATED COIL PORTION OF THE HEATER SHALL DROP INTO A 0.100 DIA RING GAUGE 3/16 in. LONG OF ITS OWN WEIGHT.
- C - UNCOATED AREA TO BE STRAIGHT AND CLEAN
- D - THERE SHALL BE NO EXPOSED BASE METAL IN THE SPECIFIED COATED AREA. PART TO BE INSPECTED USING A 10x MICROSCOPE.
- E - CUTOFF SPLITS IN TUNGSTEN WIRE SHALL NOT EXCEED 1/2 mm IN LENGTH



ATMOSPHERE	H <sub>2</sub>
TEMPERATURE	1700°C
TIME	3 MINUTES
COOL	3 MINUTES
REMARKS	FIRE AFTER EACH COAT

654814

FIGURE 4. Heater (Coated)

### 3.0 THE DIODE TEST VEHICLE

#### 3.1 Description

In this study the diode test vehicle to be used is an adaptation of a quality control vehicle presently used for cathode materials testing by the Materials and Techniques group.

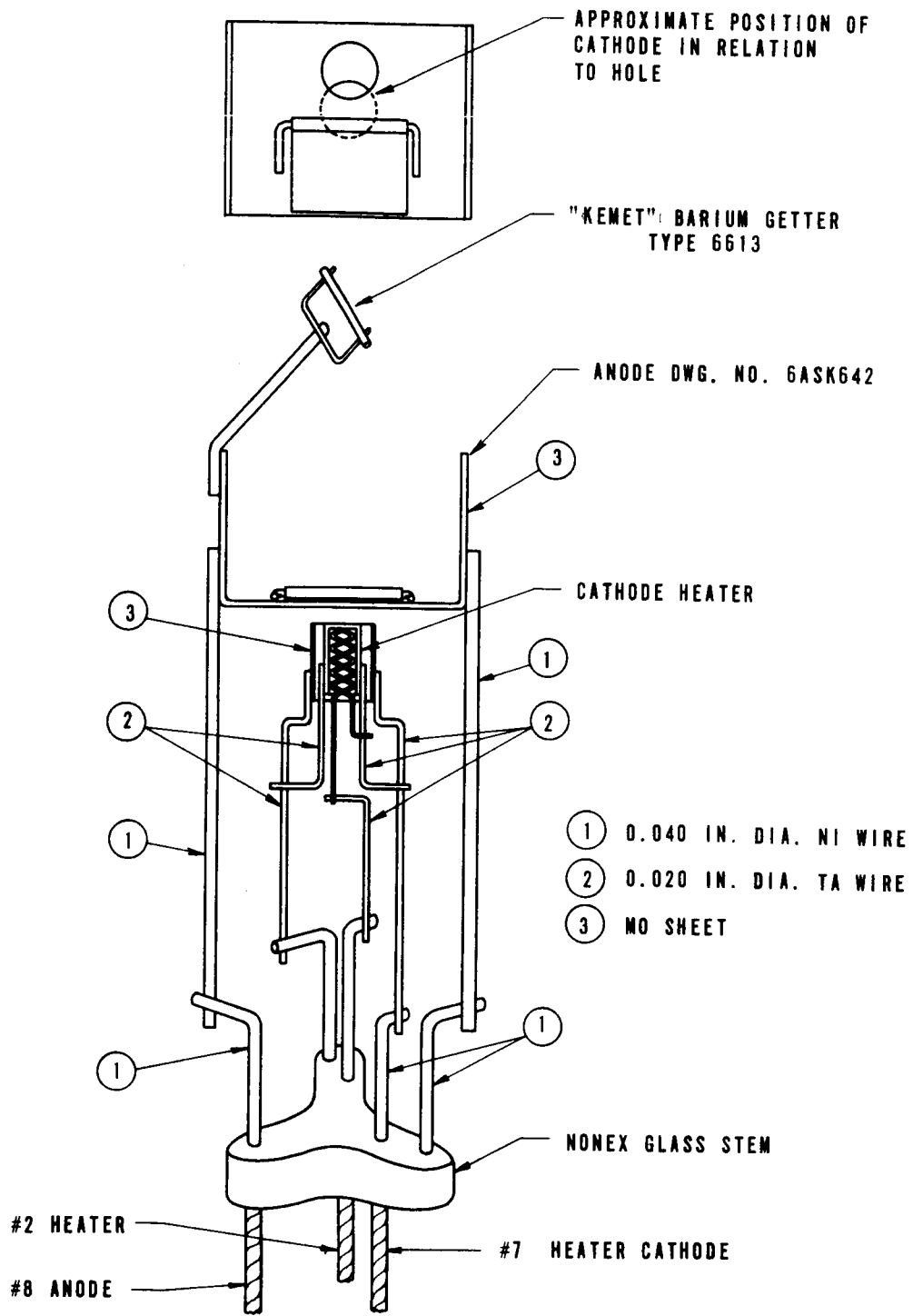
The diode is a plane-parallel type with a large, flat, winged anode, 1 in. square, and a small disc-type cathode which is the top surface of a hollow cylinder.

Construction of the proposed diode for this study is shown in Figure 5.

Original planning anticipated use of ceramic support rings for ruggedization of cathode to anode spacing. But further consideration of contamination of the cathode and anode (by degassing of the ceramic), and the probability of leakage paths across ceramics (induced by evaporated metals from the cathode structure), led to the elimination of ceramic support members.

The proposed diode will be made entirely from metals compatible for each type of cathode. Both the stem and the glass envelope are made from Corning #772 nonex hard glass. Overall dimensions of the sealed-in vehicle are 1.5 in. diameter and 4.25 in. height. The sealed-in glass vehicle is mounted into an octal bakelite socket of 1.3 in. diameter.

The cathode is heated to the desired temperature by radiation from a heater inserted into the cathode sleeve. Cathode temperature is monitored by visual observation through a hole in the anode, by optical or infrared pyrometry. A protective moveable flap is provided to limit barium evaporation to the top of the glass envelope during the life testing period. The anode is constructed large to lessen overheating of the anode during test, which can cause cathode poisoning.



654813

Figure 5. Diode Assembly for Thermionic Cathode Evaluation Study

With a vehicle of this size and construction, the maximum voltage dissipation within the vehicle will be 8 to 10 watts anode power and 10 watts heater power. Under these conditions, the effects of anode poisoning on the cathode will be minimized.

### 3.2 Construction

The diode will be constructed in an air-conditioned and humidity-controlled room.

Welds in the diode are performed by resistance welding with a stream of helium gas blowing on the weld during the operation (see Figure 6) which helps to minimize oxidation of the welded joint.

With the pore-type dispenser cathode the diode will use a radiation shield around the cathode to lower the heater power requirements.

The metals used in this diode are molybdenum, tantalum and nickel. Cleaning schedules to be used for these metals are given in Tables II, V and VI, respectively.

The glass envelope and glass stem will be cleaned in accordance with Table VII.

Construction of the diode to be used with the oxide and coated particle cathodes will be the same as that used with the pore-type dispenser cathode.

The metal to be used with this diode is nickel only and this will be cleaned by the procedure noted in Table VI.

Cathode-to-anode spacing in all cases will be 0.015 in. to 0.020 in. This will allow the diodes to operate with a maximum of 8 - 10 watts anode dissipation under the most extreme test condition that is required for this study ( $E_p = 100V$   $I_p = 0.080$  mA).



Figure 6. Resistance Welding of Diode Test Vehicle

TABLE V

## Cleaning of Tantalum Parts

Purpose

To specify a method for cleaning tantalum and tantalum parts prior to use.

Equipment

Acid-resistant sink with ventilating hood and hot and cold running water.  
Polyethylene container of suitable size for acid solution.  
Stainless steel baskets or tongs, preferably coated with a teflon type plastic.  
Clean filtered compressed air.

Materials

Hydrofluoric Acid. (concentrate reagent grade)  
Nitric Acid. (concentrate reagent grade)  
Methanol.  
Lint-free paper.

Procedure

1. Determine capacity of container by measuring (use a liter graduate) the amount of water necessary to fill it. Then pour 1/3 of this amount into empty container and mark container to indicate 1/3 volume of container. Add another 1/3 of amount necessary to fill container and mark 2/3 volume. Fill to 1/3 mark with water, add nitric acid to 2/3 volume mark, then fill with hydrofluoric acid.
2. Immerse parts for 5 seconds with gentle agitation.
3. Hold parts in fumes just above solution for 10 seconds.
4. Rinse in running cold tap water.
5. Repeat #2, 3 and 4 twice more.
6. Rinse thoroughly in running cold tap water.
7. Rinse in methanol.
8. Dry in stream of clean dry compressed air.
9. Wrap in lint-free paper.

Note

- (1) Do not handle clean parts with fingers.
- (2) Hydrofluoric acid and its containers should be handled wearing rubber gloves. Wash hands thoroughly with running water when through. Face shields should be worn while handling hydrofluoric solutions.
- (3) Shop Manual "Handling corrosive materials" applies.

TABLE VI  
Cleaning of Nickel Parts

Preparation of Solutions

1. Cleaning solution -

30% Hydrogen Peroxide	5 Parts
88% Formic Acid	10 Parts
Distilled water	80 Parts

Add the formic acid to the distilled water and then add the hydrogen peroxide.

2. Maintain bath temperature at 20°C - 25°C.
3. Change solution twice weekly.

Procedure

1. Place parts in suitable container or fixture.
2. Vapor degrease parts in permachlor.
3. Clean parts ultrasonically in Igepal solution.
4. Clean parts in solution for 8 - 9 minutes.
5. Rinse parts in overflowing tap water for 5 - 6 minutes.
6. Rinse and agitate parts in deionized water for 2 minutes minimum.
7. Rinse parts in deionized water for a minimum of 5 minutes.
8. Repeat step (7) twice, rinsing in the next cleanest tank each time.
9. Blow parts dry with filtered nitrogen.
10. Dry parts in nitrogen-fed oven (105°C-116°C) for 15 - 17 minutes.
11. Blow sample parts dry with filtered nitrogen. (Handle all parts with PVC palmed gloves).
12. Store parts in clean containers.



## TABLE VII

## Cleaning of Glass Parts

Preparation of Solution

Prepare 10% by volume - 30% hydrogen peroxide, 90% deionized water and add ammonium hydroxide to adjust pH to 11 (check with pH paper).

Procedure

- a. Boil glass piece in solution for 30 minutes.
- b. Rinse in overflowing tap water for 5 minutes.
- c. Rinse in deionized water.
- d. Dry in nitrogen-fed oven at 110°C for 15 minutes.

## 4.0 EXHAUST PROCEDURES

### 4.1 Exhaust System

Exhaust processing of all the diode test vehicles will be performed on the exhaust system shown in Figure 7. The system is located in a limited access area under the supervision of personnel of the Material and Techniques Group, who designed and built the unit originally.

Overall length of the unit is 6 feet and width is 3 feet. It is capable of maintaining a vacuum of  $10^{-9}$  torr and is equipped with an electric oven for bakeout of 6 vacuum diodes simultaneously. Each of the 6 exhaust ports has its own 8-litre Vac-Ion pump and vacuum shut-off valve. The system is backed by a K3 Kinney compound pump for rough vacuum pumping and by an oil diffusion pump (200 l/sec) and nitrogen cold trap for medium vacuum range pumping ( $10^{-3}$ - $10^{-6}$  torr).

Each glass vehicle is sealed to a glass port which is attached to a stainless steel vacuum tight flange. Each tube has its own vacuum pumping system and cannot contaminate the other tubes during the bakeout and exhaust processing step.

The unit has been in operation for over 1 year and has proved to be relatively maintenance-free and reliable for exhaust processing of vacuum tubes.

Initial loading, vacuum pumping, and 16-hour bakeout at  $450^{\circ}\text{C}$  is outlined in detail in Table VIII.

At this point, the load of tubes should be in the  $10^{-9}$  torr vacuum range and the glass parts should be relatively free of gas and water vapor.

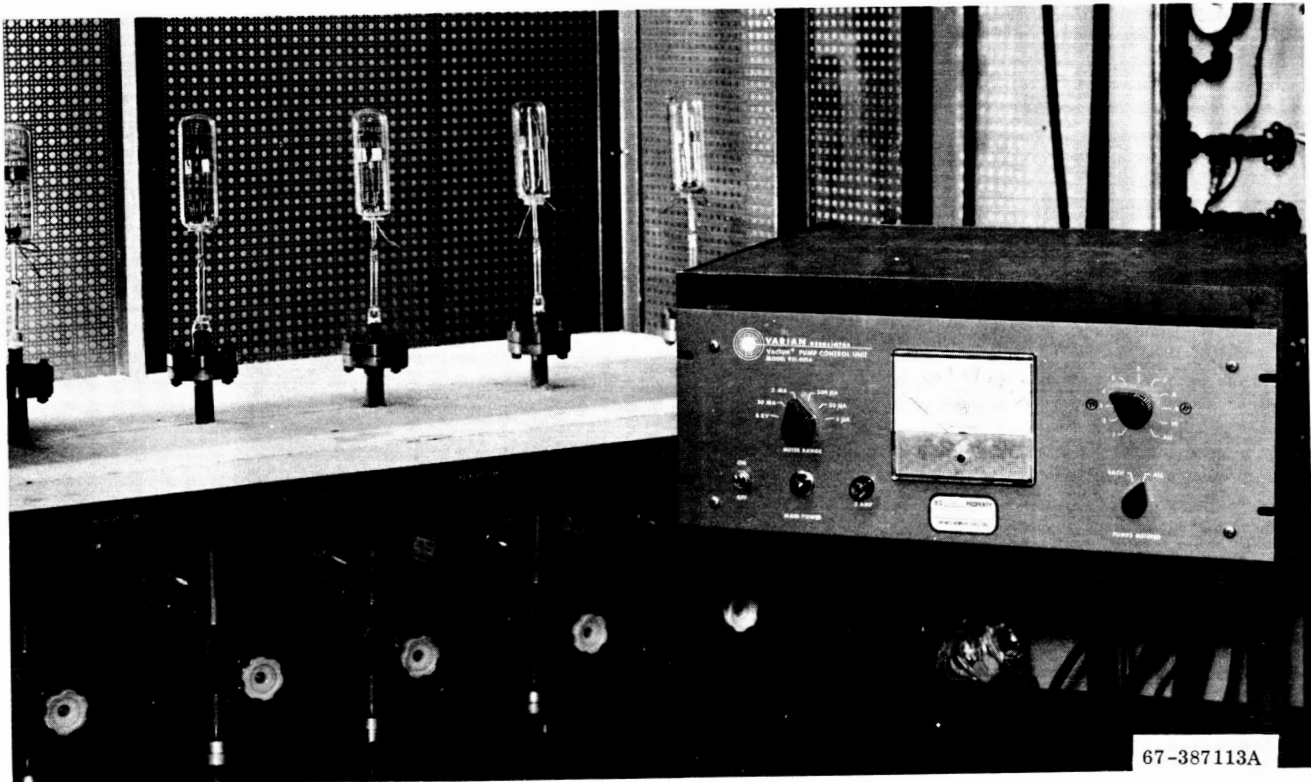


Figure 7. Exhaust System for Diode Test Vehicle  
(Cathode Laboratory)

TABLE VIII

## Exhaust Unit Operation Through Bakeout

- 1.0 Open vacuum valve leading to 6 exhaust positions. (Vac-Ion pumps are off).
- 2.0 Close valves leading to roughing and oil diffusion pump systems.
- 3.0 Open bleeder valve, cut off glass exhaust tubes and seal-on 6 glass diodes.
- 4.0 Close bleeder valve and open vacuum valve to roughing pump. Run until vacuum is at 5 microns pressure.
- 5.0 Fill liquid nitrogen cold trap, close roughing pump valve and open valve to oil diffusion pump system.
- 6.0 Pump for 2 hours or until vacuum is less than  $5 \times 10^{-6}$  torr.
- 7.0 Turn on Vac-Ion pumps and pump until Vac-Ion pumps read less than  $5 \times 10^{-5}$  torr.
- 8.0 Close off oil diffusion system and valve off each individual Vac-Ion system.
- 9.0 Pump until the 6 systems are at  $1 \times 10^{-6}$  torr.
- 10.0 Lower electric oven and heat gradually to  $450^{\circ}\text{C}$  not allowing the vacuum to exceed  $5 \times 10^{-5}$  (2-hour period).
- 11.0 Bake out diode at  $450^{\circ}\text{C}$  for 16 hours.
- 12.0 Shut off oven and cool for 2-hour period. Vacuum should be on the  $10^{-9}$  torr range.

#### 4.2 Exhaust Processing of Pore-Type Dispenser Cathodes

The pore-type dispenser cathodes are exhaust-processed according to the schedule shown in Table IX.

TABLE IX

##### Exhaust Processing of Pore-Type Dispenser Cathodes

- |    |   |
|----|---|
| a. | After bakeout, the vacuum on each diode should be on the $10^{-9}$ torr scale.  |
| b. | Slowly heat cathode to $1100^{\circ}\text{C}$ holding vacuum below $5 \times 10^{-5}$ .   |
| c. | Hold cathode at $1100^{\circ}\text{C}$ for 30 minutes.  |
| d. | After 15 minutes of cathode heating at $1100^{\circ}\text{C}$ , heat anode to $900^{\circ}\text{C}$ for 1 minute by rf heating. |
| e. | Degas Getter.   |
| f. | Turn off heater.  |
| g. | Repeat steps b - f for other 5 tubes.   |
| h. | Seal-off 6 diodes. Vacuum on each tube should be on the $10^{-9}$ torr scale.   |
| i. | Flash Getters.  |
| j. | Attach bakelite base to tubes.  |

Cathode temperature is read during the exhaust cycle by means of an optical Pyrometer (Leeds Northrup Cat. No. 8622c) aimed through the anode hole.

The main point of interest during the exhaust cycle is the degassing of the cathode and auxiliary metal parts of the tube.

Chemical generation of barium metal starts in the cathode pores and the surface of the cathode shows the presence of barium metal at the tube seal-off step.

#### 4.3 Exhaust Processing of the Barium Strontium Oxide Cathode

Procedure for exhaust processing of the barium, strontium oxide cathode is shown in Table X.

TABLE X

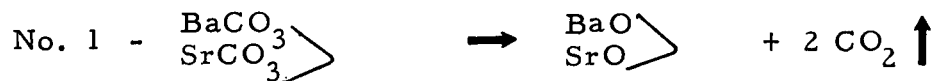
##### Exhaust Processing of Barium Strontium Oxide Cathodes

- |    |   |
|----|---|
| a. | After bakeout, the vacuum on each diode should be on the $10^{-9}$ torr scale.                                    |
| b. | Degas Getter.   |
| c. | Outgas anode at $900^{\circ}\text{C}$ for 1 minute.   |
| d. | Heat cathode to $1050^{\circ}\text{C}$ keeping pressure below $1 \times 10^{-4}$ torr (approximately 10 minutes). |
| e. | Hold 5 minutes at $1050^{\circ}\text{C}$ .  |
| f. | Drop cathode temperature to $950^{\circ}\text{C}$ and hold 10 minutes.  |
| g. | Turn off heater.  |
| h. | Repeat steps (b) - (g) for other 5 diodes.  |
| i. | Seal off 6 diodes. Vacuum on each tube should be on the $10^{-9}$ torr scale.                                     |
| j. | Flash Getter.   |
| k. | Attach bakelite bases to tubes.   |

The chemical reactions occurring during the exhaust process of this cathode are entirely different from those of the pore-type dispenser cathode.

In addition to accomplishing the degassing of auxiliary metal parts of the diode, other gaseous end products are produced by the chemical reactions for producing barium metal in the cathode system.

The initial step in cathode processing is the conversion of the cathode coating to the oxides.

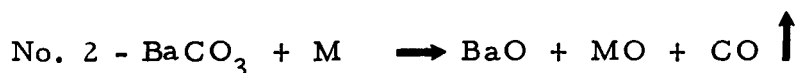


This is accomplished by heating the cathode as rapidly as possible to an elevated temperature (1050°C) and pumping off the carbon dioxide gas through the vacuum pumping system.

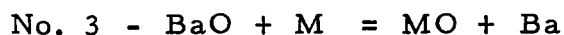
The second step in the processing of the cathode is the generation of barium metal within the oxide coating body.

When the cathode is heated, minute quantities of reducing impurities (i.e., zirconium) will diffuse to the surface of the cathode at the interface of the nickel body and the oxide coating. Zirconium metal will react chemically with either the alkaline earth carbonates or alkaline earth oxide; the reaction is dependant on the state of decomposition of the cathode coating.

If the reducing impurity sees a carbonate, the reaction will be as follows, in the case of barium carbonate:



If the reducing impurity sees an oxide, the reaction will be as follows in the case of barium oxide:



In summarizing the barium generating reactions, the alkaline earth carbonates should be reduced to the oxides as rapidly as possible to prevent the deleterious reactions from taking place (Reaction No. 2).

During the lifetime operation of the oxide cathode, we have a system generating barium continuously. The reducing impurity diffuses to the nickel cathode surface, reacts with barium oxide, produces free barium metal, and eventually evaporates from the coating to the nearest cool surface.

The current loading capabilities of the oxide cathode are dependant on the number of barium atoms present within the oxide coating. The concentration of barium within the coating will decrease during the lifetime of the tube and will eventually deplete itself. The current capabilities of the oxide cathode will show a gradual decay and eventual failure during the lifetime operation of the cathode.

#### 4.4 Exhaust Processing of the Coated Particle Cathode

Procedure for exhaust processing of the coated particle cathode is shown in Table XI.

TABLE XI

##### Exhaust Processing of Coated Particle Cathodes

- |    |   |
|----|---|
| a. | After bakeout, the vacuum on each diode should be on the $10^{-9}$ torr scale.  |
| b. | Degas Getter.   |
| c. | Outgas anode at $900^{\circ}\text{C}$ for 1 minute.   |
| d. | Heat cathode slowly to $850^{\circ}\text{C}$ keeping pressure below $5 \times 10^{-6}$ torr (approximately 90 minutes). |
| e. | Raise cathode temperature to $1050^{\circ}\text{C}$ rapidly and hold for 20 minutes.                                    |
| f. | Draw 50 miliamperes of current to anode and hold for 3 minutes.   |
| g. | Drop cathode temperature to $950^{\circ}\text{C}$ .   |
| h. | Repeat step (f).  |
| i. | Drop cathode temperature to $850^{\circ}\text{C}$ .   |
| j. | Repeat step (f).  |
| k. | Turn off heater.  |
| l. | Repeat steps (b) - (k) for other 5 diodes.  |
| m. | Seal off 6 diodes. Vacuum on each tube should be on the $10^{-9}$ torr scale.   |
| n. | Flash Getter.   |
| o. | Attach bakelite bases to tubes.   |



Chemical reactions occurring during this exhaust processing are the same as in the case of the barium, strontium oxide cathode.

The cathode is processed very slowly to 850°C to convert the alkaline earth carbonate to the oxides and to minimize the oxidation of the nickel particles in the body of the coating.

The barium-producing mechanism is the same as in the case of the oxide cathode.

Both types of cathodes should generate the same amount of barium metal and have approximately the same life capabilities and the same failure mechanism.

The only chemical mechanism present that will reduce the free barium supply is the oxidation of the nickel member of the coating and the subsequent reduction of the nickel oxide by the free barium present in the coating to form barium oxide.

## 5.0 ELECTRICAL TEST PROCEDURES

In order to comply with electrical test procedures as shown in Table XII as well as and the current density level specification as shown in Figure 8, a series of test diodes have been constructed and are undergoing preliminary electrical testing.

Cathodes were constructed according to the specification submitted in Section 2.0, Thermionic Cathode Design. The diodes were constructed and exhausted according to the specifications shown in Section 3.0, Diode Test Vehicle and Section 4.0, Exhaust Procedures.

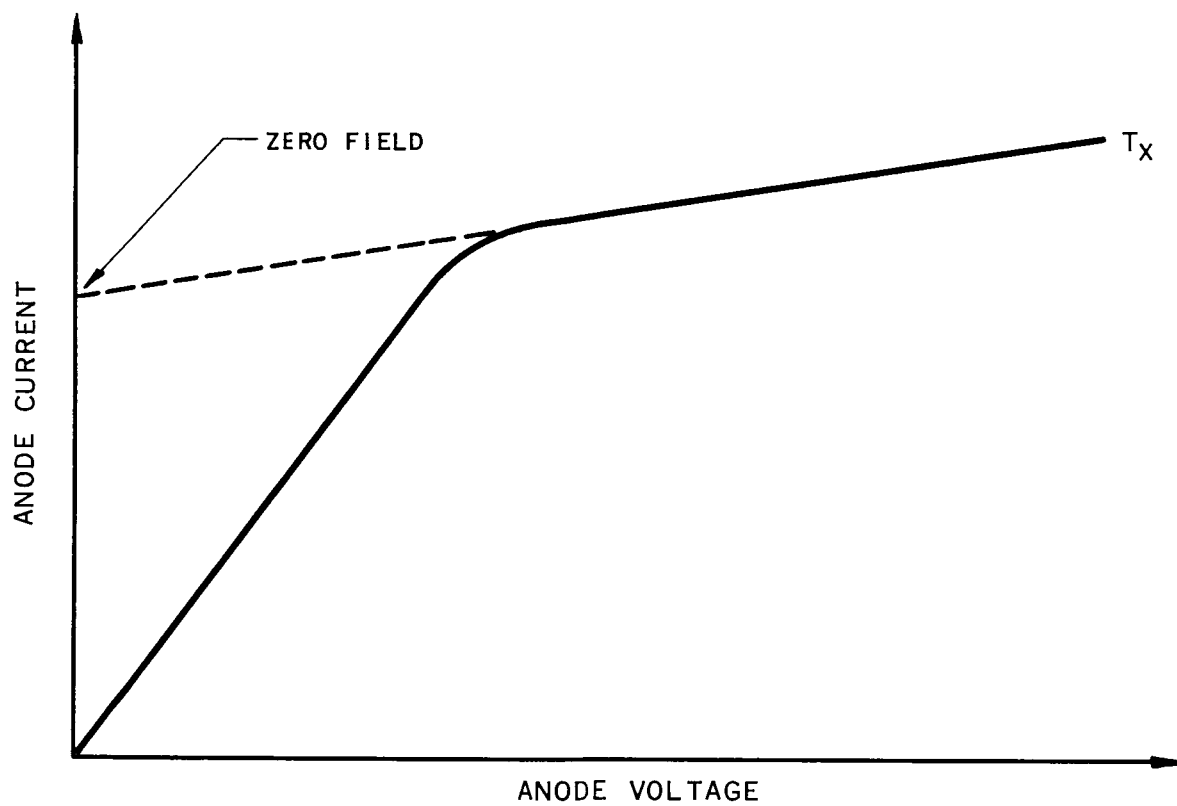
Diodes were subjected to a preliminary life burning procedure on the electrical test rack shown in Figure 9.

The diodes were burned at  $1100^{\circ}\text{C}$ , in the case of the pore-type dispenser cathode, and at  $850^{\circ}\text{C}$ , in the case of the oxide and coated particle cathode. In all cases the anode voltage was set at 50 Vdc.

During the course of life burning of the diodes, they were checked for perveance and zero field anode current according to the specification shown Figure 8. This was accomplished by variation in cathode temperature and in plate voltage on the above-mentioned life burning rack.

During various intervals of life burning, the diodes were tested for dip temperature by the schematic diagram shown in Figure 10.

The diode was set into a socket with the anode flap in the open position. Cathode temperature was read and set by means of an optical pyrometer. Then the infrared pyrometer was pointed on the cathode and was calibrated and set to read the optical temperature from the prescribed temperature to room temperature on the X-axis or abscissa. The anode current was set on the Y-axis or ordinate to read 10 milliamperes of current per centimeter.



654815

FIGURE 8. Current Density Level (JPL Specification)

TABLE XII  
Electrical Test Procedures (JPL Specification)

Diode Selection				Life-Test Operation			
	Req'd Units	Life Test Temp	Zero Field Current Dens ma/sq cm	Req'd Units	Current Density ma/sq cm	Req'd Units	Current Density ma/sq cm
Oxide Cathodes	4	T <sub>1</sub>	250	2	75	2	150
	4	T <sub>2</sub>	500	2	150	2	300
	4	T <sub>3</sub>	750	2	225	2	450
	4	T <sub>4</sub>	1000	2	300	2	600
CP Cathodes	4	T <sub>1</sub>	345	2	138	2	275
	4	T <sub>2</sub>	690	2	275	2	550
	4	T <sub>3</sub>	1035	2	415	2	830
	4	T <sub>4</sub>	1380	2	550	2	1100
Dispenser Cathodes	4	T <sub>1</sub>	400	2	200	2	400
	4	T <sub>2</sub>	800	2	400	2	800
	4	T <sub>3</sub>	1200	2	600	2	1200
	4	T <sub>4</sub>	1600	2	800	2	1600

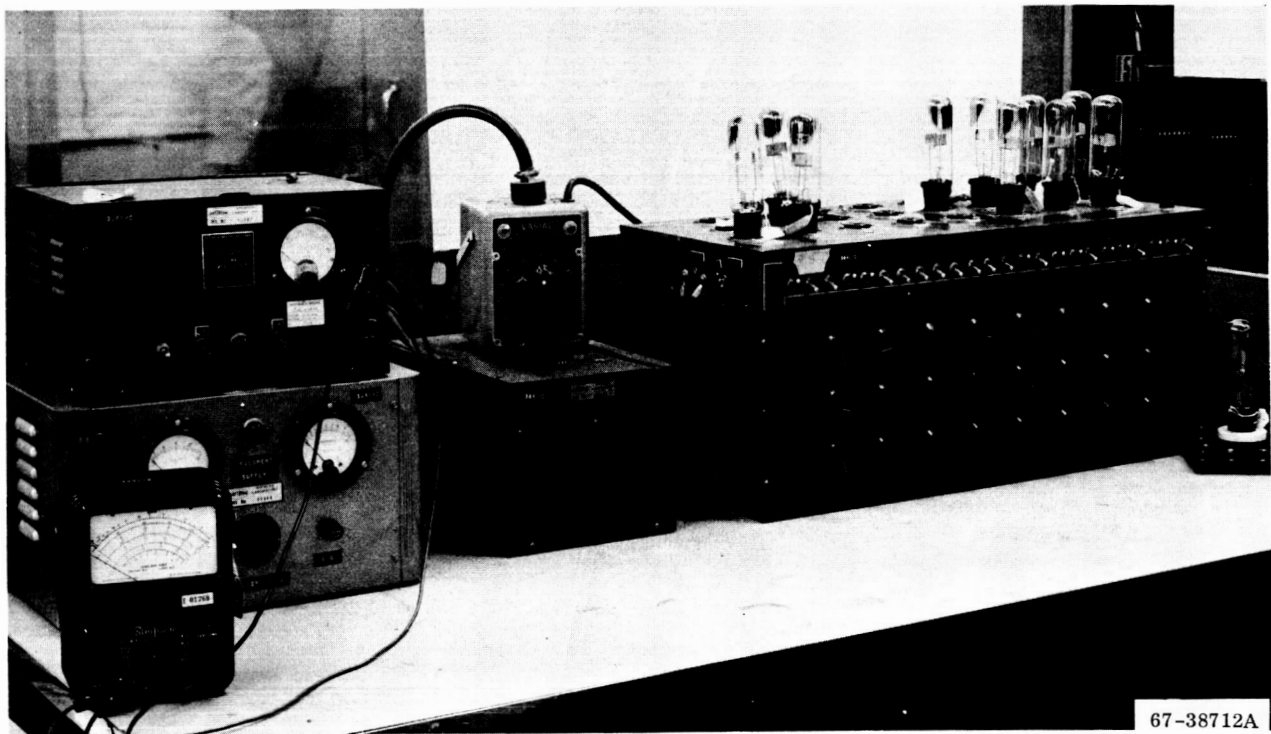
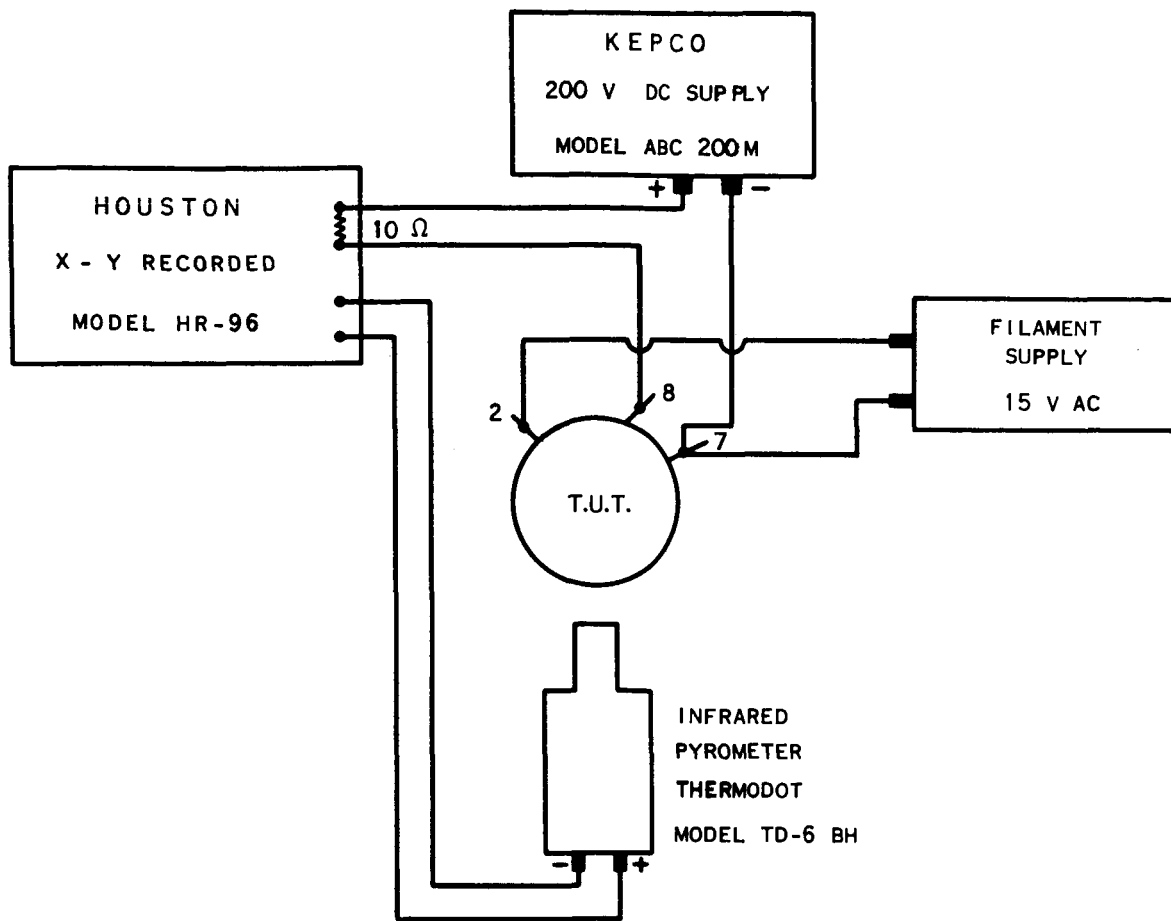


Figure 9. Diode Electrical Test Rack  
(Cathode Laboratory)



T.U.T. - TUBE UNDER TEST  
 PIN 2 - HEATER  
 PIN 7 - HEATER, CATHODE  
 PIN 8 - ANODE

654816

Figure 10. Dip Test of Diode Vehicles

In the following discussion we will present the perveance plots and dip temperature readings for various diodes that are presently under test for compliance with the requirements of this study.

### 5.1 Pore-Type Dispenser Cathodes

Twelve diodes have been constructed according to the manufacturing procedures for pore-type dispenser cathodes.

The life burning results are shown in Table XIII. It should be noted that the diodes vary in anode-to-cathode spacing from 0.015 in. to 0.025 in. The diodes were life tested at 1100°C and 50 Vdc anode voltage. Temperature and anode voltage were not varied to comply with the required test specifications because of the non-availability of the proper test equipment at the start and life burning of the diodes.

Current readings for the diodes are shown in Table XIII and represent a typical life burning characteristic for similar quality control diodes up to 900 hours of life burning.

The perveance plots for 2 diodes are shown in Figures 11 and 12. It can be seen that the zero field current can be extrapolated up to 50 mA of anode current ( $1.0 \text{ amp/cm}^2$ ) very readily. Difficulty was experienced above current densities of  $1.0 \text{ ampere/cm}^2$  to  $1.6 \text{ amperes/cm}^2$ . At anode dissipation above 12 watts, the anode would overheat and the diode current would run away very rapidly.

Dip test plots recorded on the X - Y recorder are shown in Figures 13 and 14 for two different points of life burning.

Dip temperature at three different current levels is shown by extrapolation of the temperature-limited portions of the curve to the initial current levels.

It is anticipated that the operating temperatures for dispenser-type cathodes at current levels above  $1.0 \text{ ampere/cm}^2$  can be predetermined from the extrapolation of dip temperatures.

TABLE XIII

Life Burning - Pore-Type Dispenser Cathodes

1100°C - 50 V  $E_P$ 

Tube No.	Spacing (inches)	Hours					
		0	22	94	238	564	996
P507	0.025	15.0	14.0	14.4	15.0	16.9	16.5
K31	0.025	10.4	12.4	12.4	12.4	12.8	12.0
C98	0.025	14.2	14.0	13.9	14.7	14.2	14.8
G90	0.025	19.0	12.9	13.1	12.1	13.0	12.4
C18	0.025	18.0	10.9	12.3	13.0	12.2	11.9
X10	0.025	22.0	21.0	17.0	18.0	19.8	19.9

Tube No.	Spacing (inches)	Hours				
		0	24	137	477	903
P527	0.020	13.4	15.2	15.9	15.7	15.7
X59	0.020	9.8	17.0	15.9	20.6	20.6
C63	0.020	24.0	24.0	23.0	23.0	24.0
None	0.015	36.0	29.0	30.0	31.0	31.0
A10	0.015	31.2	23.9	22.1	28.4	28.4
A19	0.015	40.0	34.7	51.0	56.0	55.0

Readings are in milliamperes of anode current.



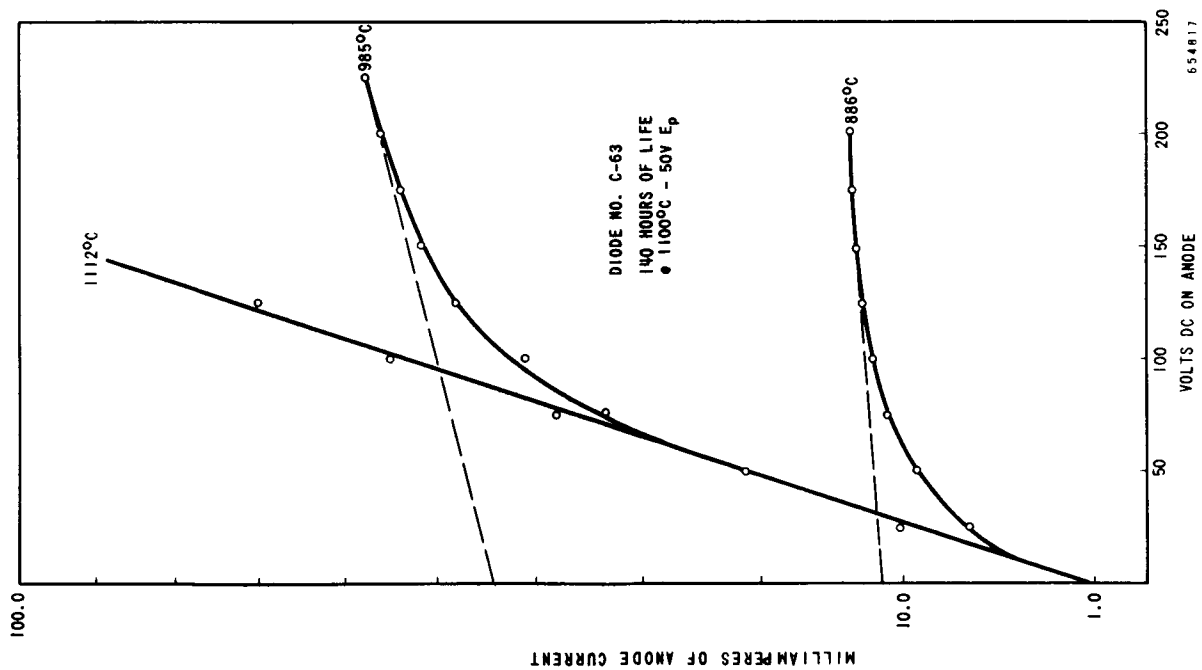


Figure 11. Pore-Type Dispenser Cathode  
Diode No. C-63

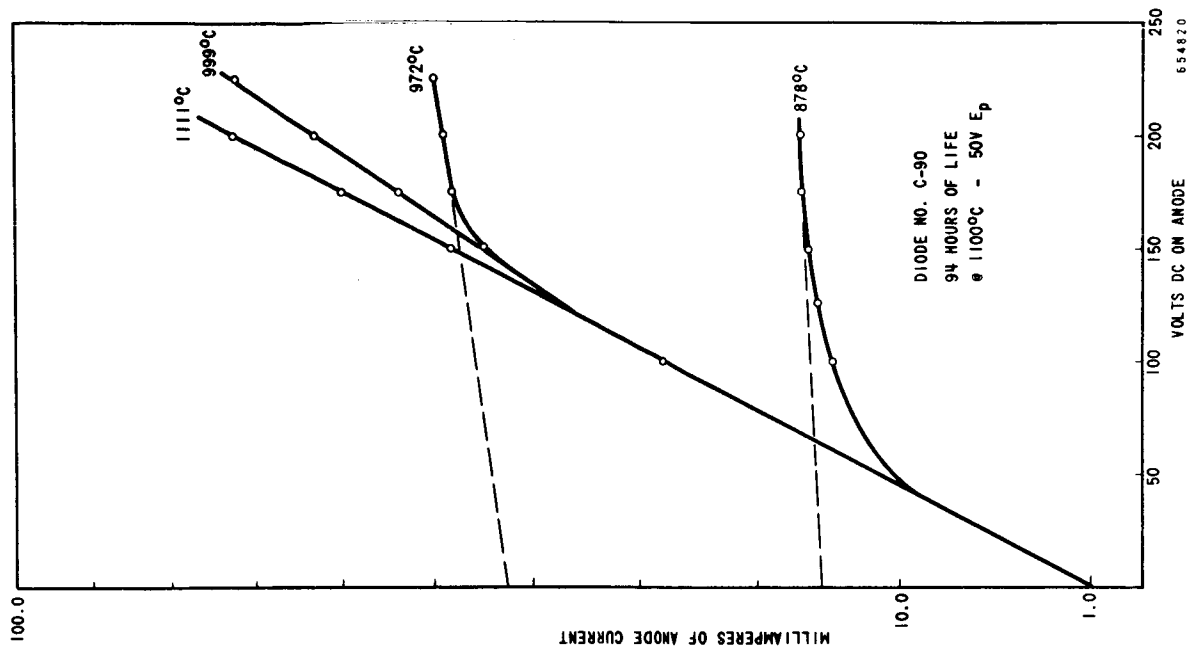


Figure 12. Pore-Type Dispenser Cathode  
Diode No. C-90

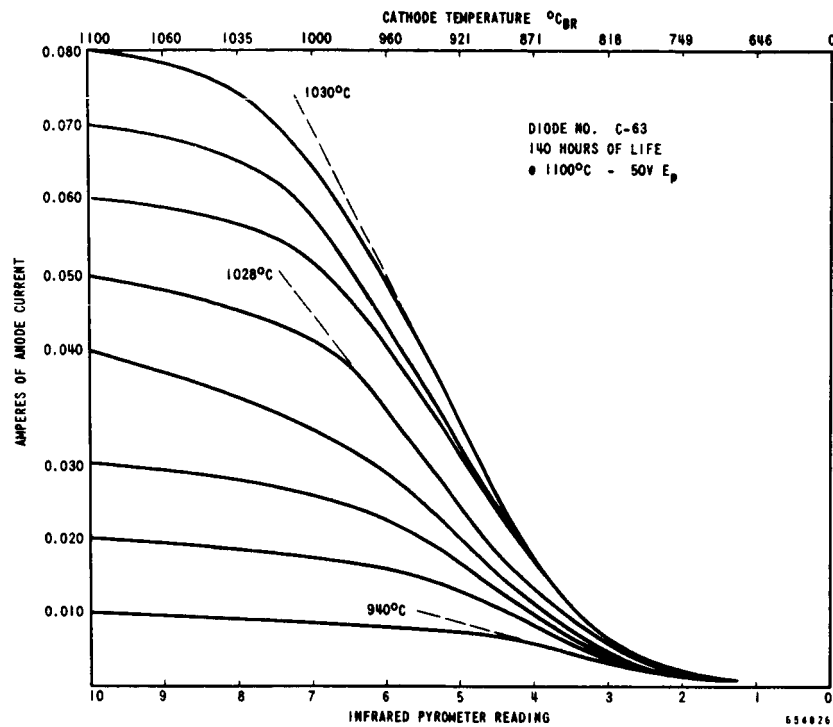


Figure 13. Pore-Type Dispenser Cathode

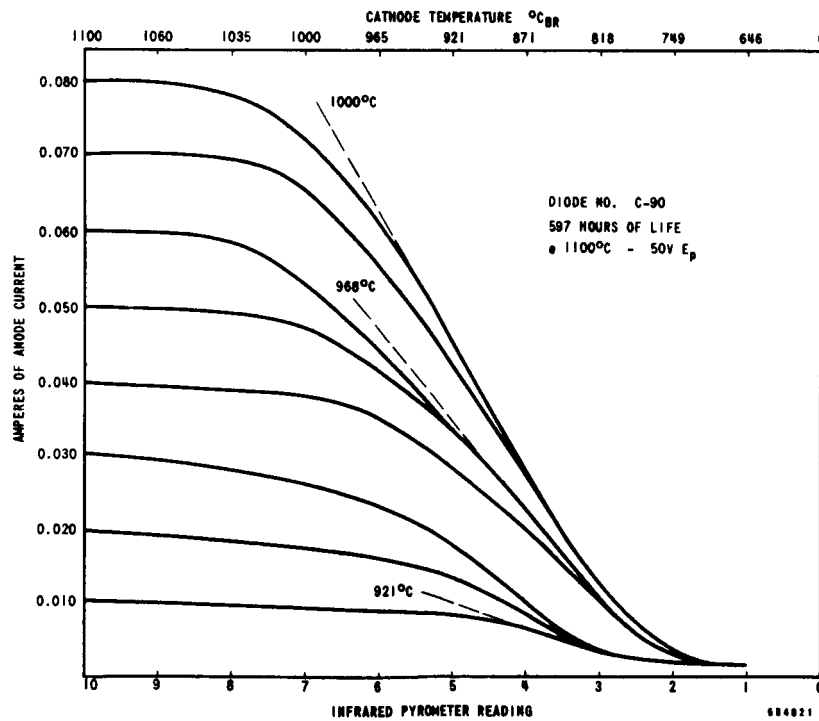


Figure 14. Pore-Type Dispenser Cathode

## 5.2 Standard Barium Strontium Oxide Cathode

Five diodes have been constructed using the procedures outlined for the standard barium strontium oxide cathode.

The cathodes were burned on life at 825 - 850°C with the anode voltage at 50 Vdc. Current readings for 500 hours of life burning are shown in Table XIV. It should be noted that the current levels spread from 24 to 38 mA due to the change in spacing from 0.015 in. to 0.020 in. The zero hour current levels are very low in comparison to the readings noted for the pore-type dispenser cathodes. This cathode takes approximately 50 hours to age in because of the limits of exhaust processing and activation schedules. This is necessary to conserve the barium supply for long life burning.

Figure 15 shows the perveance plots for the 5 diodes at 825°C - 850°C. Saturated current levels range from 30 - 50 mA, which represents 0.48 - 0.64 amperes/cm<sup>2</sup> (Area = 0.0785 cm<sup>2</sup>).

Figures 16 and 17 show the dip temperature plots for one diode at 850°C and 900°C.

It is anticipated that this oxide cathode diode will meet the life test requirements (up to 0.60 amp/cm<sup>2</sup>) at 850°C.

At this time, 6 more diodes using oxide cathodes on 0.1% Zr - Ni are being exhausted and tested for electrical parameters.

## 5.3 Coated Particle Cathode

Five diodes have been constructed using the procedures that have previously been mentioned for the coated particle cathode.

These diodes were randomized with the previously-mentioned oxide cathode diodes and were exhausted and tested together with the oxide cathode diodes.

TABLE XIV

Life Burning - Oxide and Coated Particle Cathodes

850°C - 50 V  $E_P$ 

<u>Tube No.</u>	<u>Spacing (inches)</u>	Hours			
		0	47	280	502
Oxide -1	0.015 - 0.020	7.0	18.0	40.3	35.0
Oxide -3	0.015 - 0.020	6.4	39.0	37.0	34.9
CP -4	0.015 - 0.020	3.9	20.0	26.0	26.4
CP -5	0.015 - 0.020	3.9	22.0	34.0	33.0
CP -6	0.015 - 0.020	2.0	8.8	23.2	20.7

<u>Tube No.</u>	<u>Spacing (inches)</u>	Hours			
		0	104	232	434
CP -7	0.015 - 0.020	1.0	44.1	43.2	50.0
CP -9	0.015 - 0.020	1.0	19.0	18.0	24.2
Oxide -10	0.015 - 0.020	1.4	24.2	23.9	24.4
Oxide -11	0.015 - 0.020	7.9	35.6	36.0	38.2
Oxide -12	0.015 - 0.020	4.7	23.2	24.0	23.2

Readings are in milliamperes of anode current.

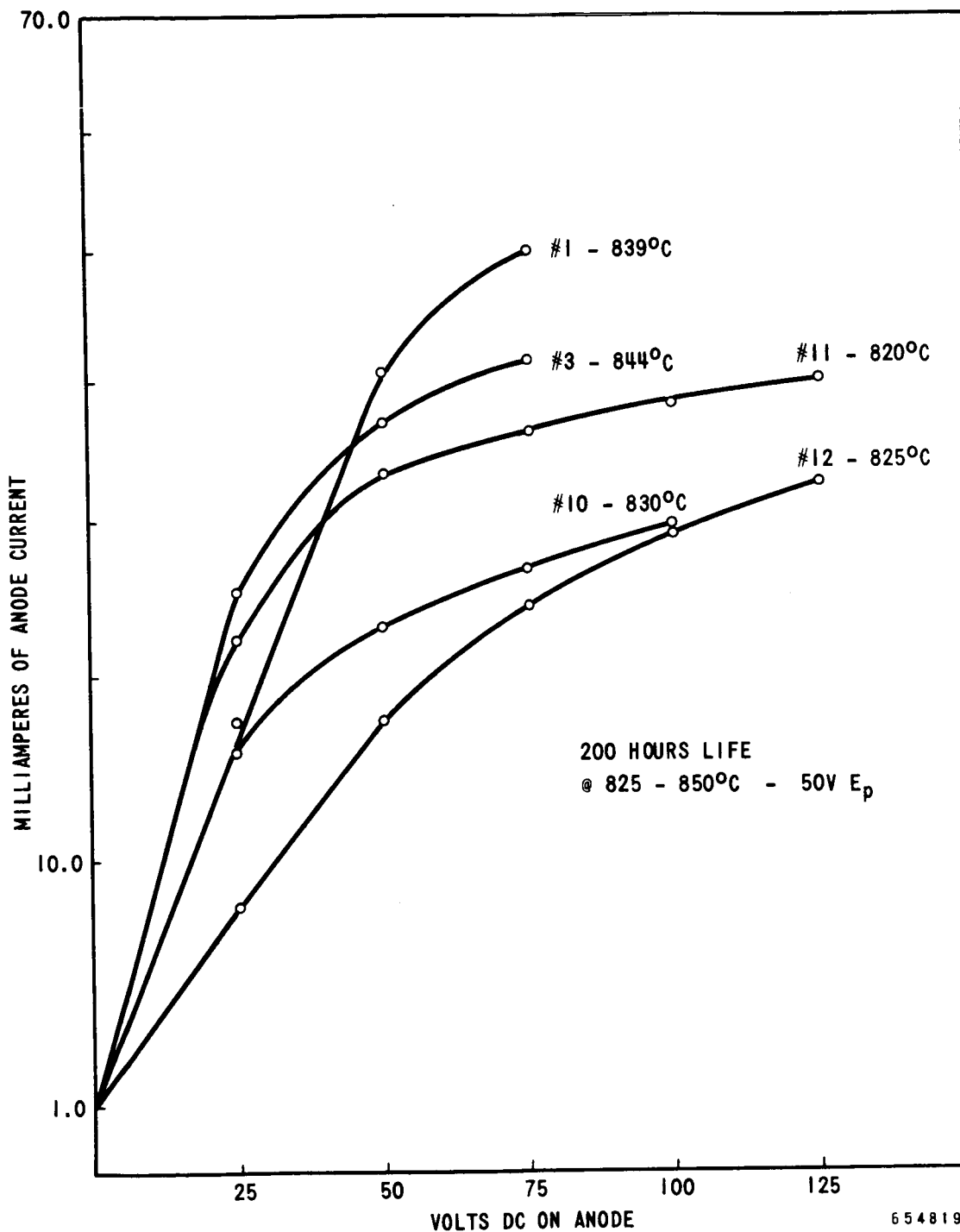


Figure 15. Oxide Cathode

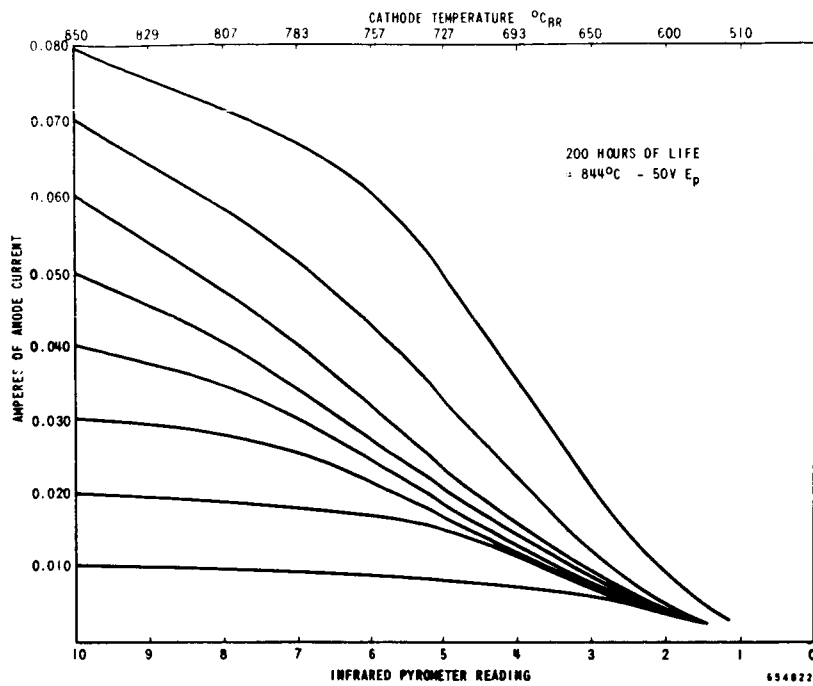


Figure 16. Oxide Cathode Diode No. 3

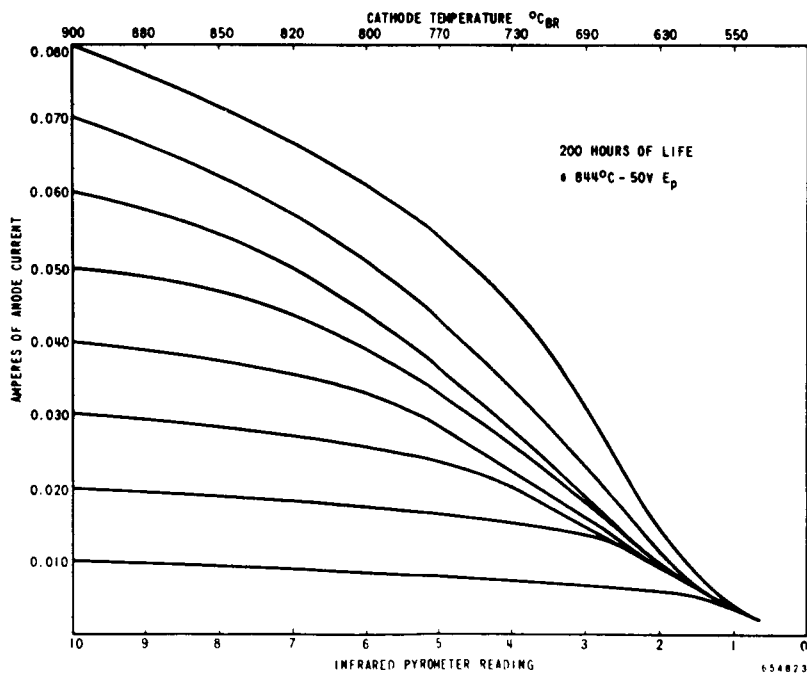


Figure 17. Oxide Cathode Diode No. 3

The cathodes were life-tested under the same conditions noted above for the oxide cathodes.

Life burning results (see Table XIV) show the same type of current spread as the oxide cathodes and the zero hour current levels are also low. This cathode will take approximately 50 - 150 hours of aging under the stated conditions to be satisfactory for life testing.

Figure 18 shows the perveance plots for the 5 diodes at 825°C - 850°C. The spread in anode currents is from 0.32 to 0.70 amperes/cm<sup>2</sup> (Area = 0.0785cm<sup>2</sup>) in the temperature saturation range.

Figures 19 and 20 show the dip tests for one diode at 850°C and at 900°C starting temperature. It can be seen that the cathode is very inactive at 850°C under these particular test procedures. The dip tests at 900°C show good performance for meeting the current requirements (max = 1.1 amp/cm<sup>2</sup>) for this study.

Six more coated particle cathode diodes are being exhausted and tested together along with the oxide cathode diodes. These next 6 diodes will be pretested with the cathode temperature at 900°C.

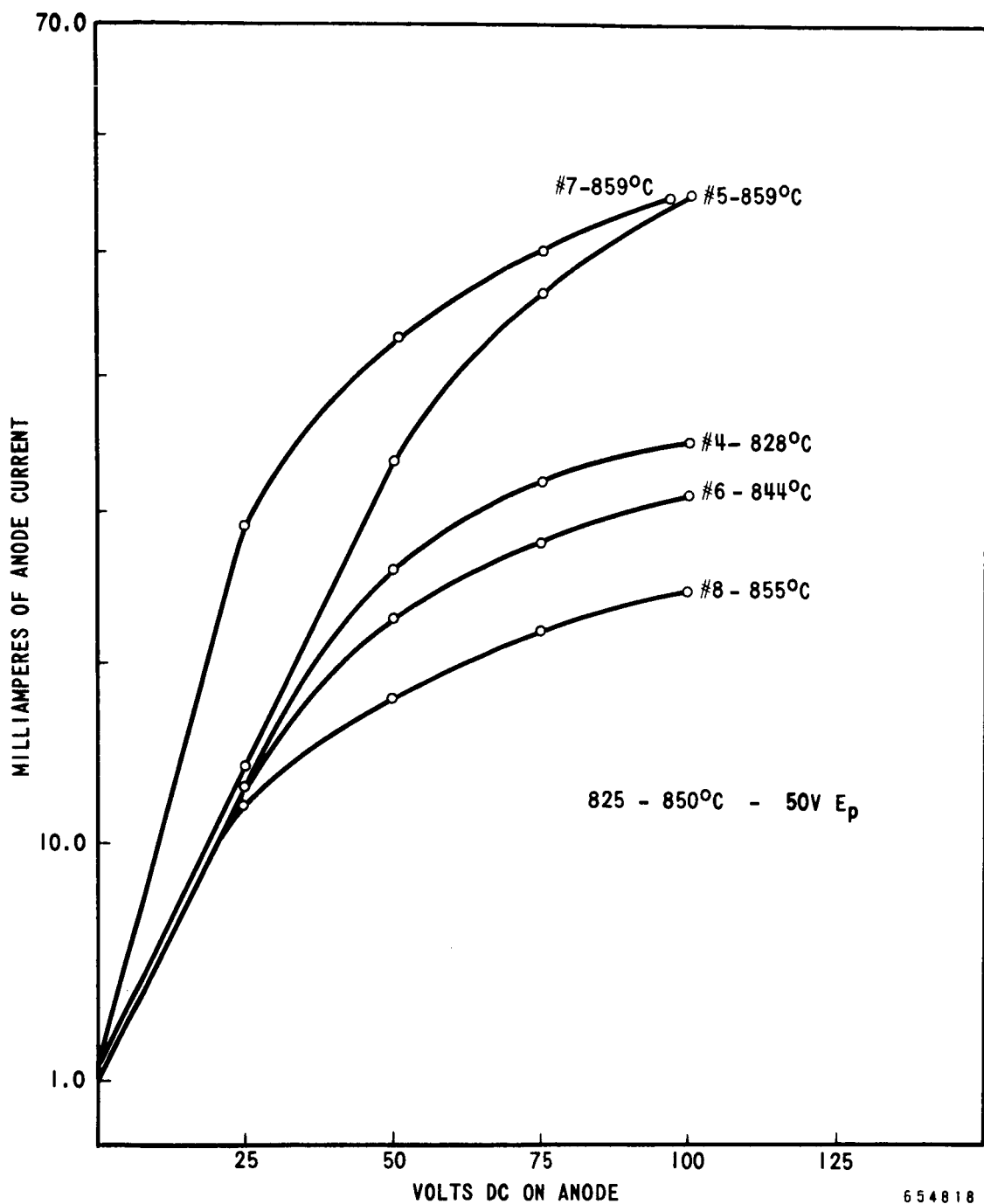


Figure 18. Coated Particle Cathode



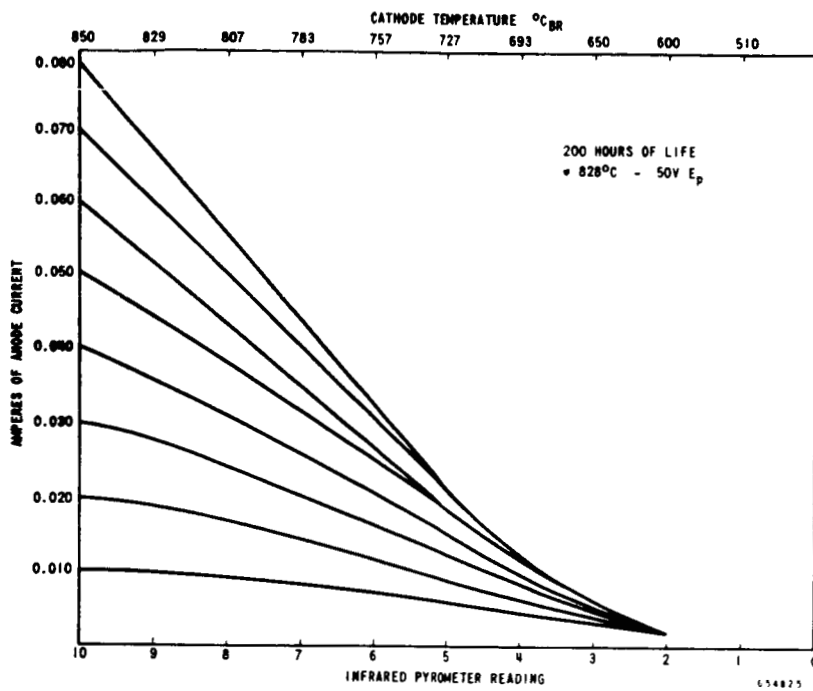


Figure 19. Coated Particle Cathode Diode No. 4

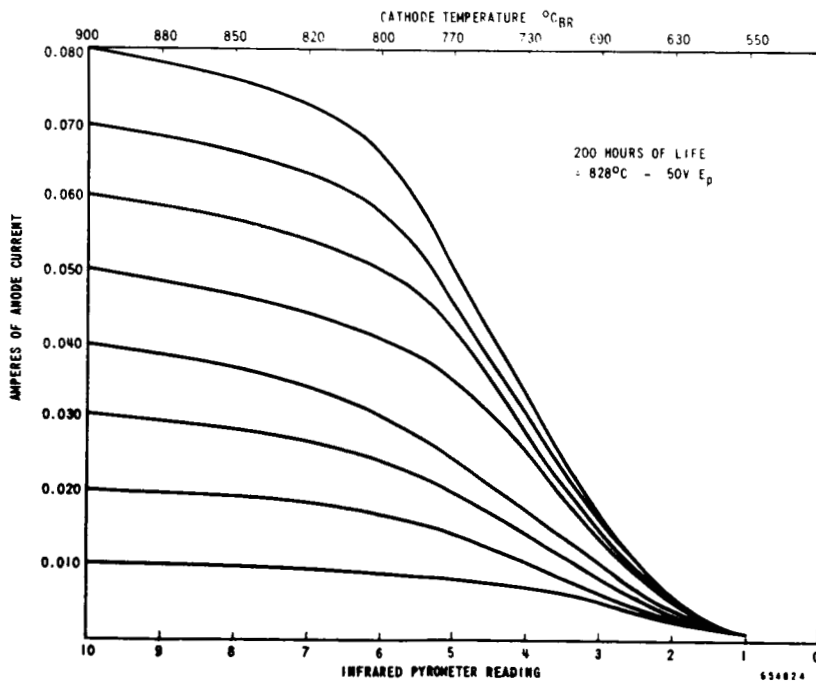


Figure 20. Coated Particle Cathode Diode No. 4

## 6.0 LIFE TEST EQUIPMENT

In anticipation of the life test requirements for this thermionic cathode study, a life test rack was designed by Raytheon personnel and was constructed by the Cober Electronics Company of Stanford, Connecticut.

The life test equipment was delivered to the Material and Techniques Group on May 31, 1967.

At present, the various diodes that were constructed for preliminary electrical testing, have been placed on the life rack to test out the stability and voltage control of the equipment. These diodes will be left on this rack for life test burning until the sockets are needed for the eventual diode tests required for this study.

A photograph of the equipment is shown in Figure 21.

The cathode life test rack, Cober Model No. 1369, has 48 test positions with each position having its own controls.

Sockets are divided into 3 banks of 16 sockets each. Each bank has its own regulated filament supply which can deliver 24 volts at 20 amperes ac to each bank. Each test position has a 4-ohm, 50 watt rheostat to adjust filament current for control of cathode temperature. The cathode temperature can be varied up to 100°C on each bank over the 800°C - 1100°C range setting on the particular bank.

A variable constant plate voltage is supplied to each socket in the range of 25 to 200 V dc.

Each socket has its own module for adjustment of plate voltage which feeds from two Technipower solid-state model L-100.0 - 6.0 M regulated power supplies.

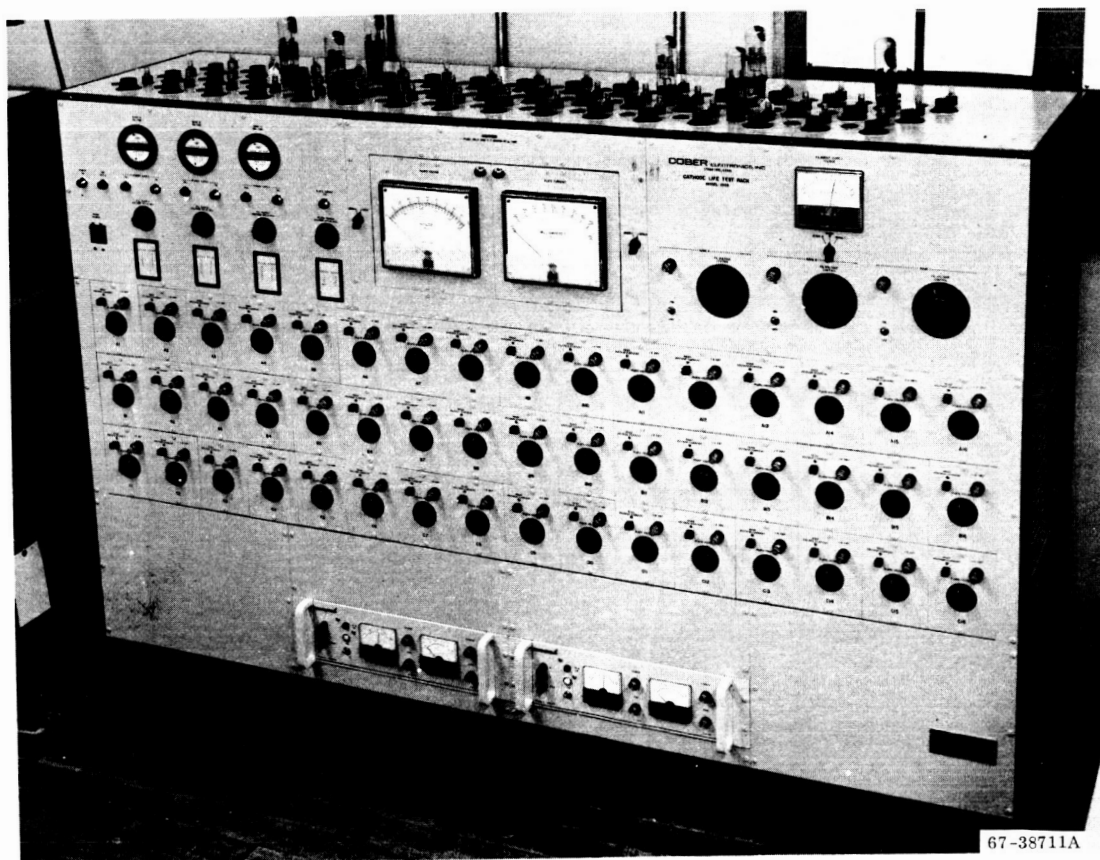


Figure 21. Cathode Life Test Rack  
(Cathode Laboratory)

The constant voltage setting on each socket is monitored by a calibrated 7-1/2 inch square tautband meter, Weston Model 1971. It has a sensitivity of 20,000 ohms per volt with internal multipliers permitting two ranges, 0 to 100 Vdc and 0 - 250 Vdc.

The plate current on each socket is monitored by a calibrated 7-1/2 inch square, tautband, 50-microampere meter, Weston Model 19171. It is provided with multipliers and shunts to provide ranges of 0 to 100 mA and 0 to 25 mA dc.

Preliminary indications show the test equipment to be satisfactory for the life test requirements of this 2 year study.

## 7.0 PLANS FOR THE SECOND QUARTER

The second quarter of this study which will extend from July 1 to September 30, 1967 will include the following work schedule:

- A - Continue stabilization tests on life test equipment.
- B - Continue and complete pretesting of oxide cathode and coated particle cathode diodes.
- C - Manufacture final cathodes (three types) for diodes.
- D - Construct 48 diodes, consisting of 16 for each type of cathode.
- E - Pre-age and test each diode for selection of test parameters for this study.
- F - Start life testing of all diodes as of October 1, 1967.